

AD-A173 356

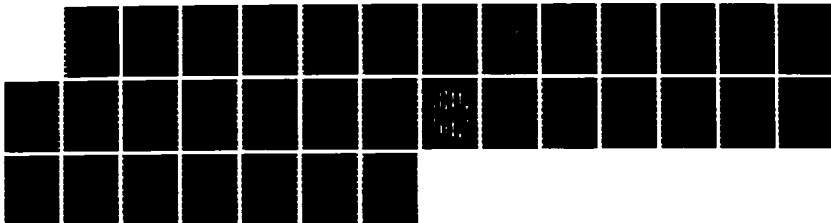
AN INVESTIGATION OF THE STRUCTURE AND HIGH TEMPERATURE
MECHANICAL PROPERT (U) STANFORD UNIV CA DEPT OF
MATERIALS SCIENCE AND ENGINEERING W D NIX SEP 86
AFOSR-TR-86-1002 AFOSR-81-0022

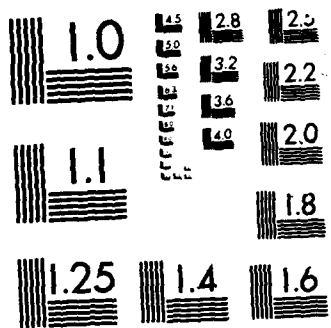
1/1

UNCLASSIFIED

F/G 11/6

NL





2

Final Scientific Report
(For the Period 1 October 1980 to 30 September 1985)
AFOSR GRANT NO. 81 - 0022

**AN INVESTIGATION OF THE STRUCTURE AND HIGH TEMPERATURE
MECHANICAL PROPERTIES OF OXIDE DISPERSION STRENGTHENED
ALLOYS**

Submitted to

Department of the Air Force
Directorate of Electronic and Material Sciences
Air Force Office of Scientific Research
Bolling Air Force Base, Building 410
Washington, D.C. 20332

Attention: Dr. Alan H. Rosenstein

Submitted by

Professor William D. Nix, Principal Investigator
Department of Materials Science and Engineering
Stanford University, Stanford California 94305

September, 1986

This research was supported by the Air Force Office of Scientific Research (AFOSC) under Grant No. AFOSR 81 - 0022. Approved for public release; distribution unlimited.

Qualified requesters may obtain additional copies from the Defense Documentation Center; all others should apply to the ~~Clearing House for Federal Scientific and Technical Information.~~

TECHNICAL INFORMATION CENTER.
NATIONAL TECHNICAL INFORMATION SERVICE.

DTIC
ELECTE
OCT 22 1986
S B

AD-A173 356

DTIC FILE COPY

REPORT DOCUMENTATION PAGE

1a. REPORT SECURITY CLASSIFICATION <u>Unclass.</u>			1b. RESTRICTIVE MARKINGS <u>None</u>		
2a. SECURITY CLASSIFICATION AUTHORITY			3. DISTRIBUTION/AVAILABILITY OF REPORT <u>Open Distribution / Unlimited</u>		
2b. DECLASSIFICATION/DOWNGRADING SCHEDULE					
4. PERFORMING ORGANIZATION REPORT NUMBER(S)			5. MONITORING ORGANIZATION REPORT NUMBER(S) AFOSR-TR. 86-1002		
6a. NAME OF PERFORMING ORGANIZATION <u>STANFORD UNIV.</u>	6b. OFFICE SYMBOL (If applicable) <u>MSE</u>		7a. NAME OF MONITORING ORGANIZATION <u>AFOSR/NE</u>		
6c. ADDRESS (City, State and ZIP Code) <u>STANFORD, CALIF. 94305</u>			7b. ADDRESS (City, State and ZIP Code) <u>Bolling AFB, DC 20332</u>		
8a. NAME OF FUNDING/SPONSORING ORGANIZATION <u>AFOSR</u>	8b. OFFICE SYMBOL (If applicable) <u>NE</u>		9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER <u>AFOSR-81-0022</u>		
8c. ADDRESS (City, State and ZIP Code) <u>Bolling AFB, DC 20332</u>			10. SOURCE OF FUNDING NOS.		
			PROGRAM ELEMENT NO. <u>61102F</u>	PROJECT NO. <u>2306</u>	TASK NO. <u>A1</u>
			WORK UNIT NO. <u>—</u>		
11. TITLE (Include Security Classification) <u>USE TITLE ON COVER</u> <u>(U) Structure and High Temp. Materials</u>					
12. PERSONAL AUTHOR(S) <u>Wm. D. Nix</u> <u>Properties of 6061 Alloys</u>					
13a. TYPE OF REPORT <u>FINAL</u>	13b. TIME COVERED FROM <u>1 Oct 80</u> TO <u>30 Sept 85</u>		14. DATE OF REPORT (Yr., Mo., Day) <u>Sept 1986</u>		15. PAGE COUNT <u>17</u>
16. SUPPLEMENTARY NOTATION					
17. COSATI CODES			18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)		
FIELD <u>11.06</u>	GROUP	SUB. GR.	<u>Aluminum Alloys</u> <u>High Temp. Aluminum Alloys</u> <u>Oxide Dispersion Strengthened</u> <u>Nickel Alloys</u> <u>17A-754</u> <u>MA-6000</u>		
19. ABSTRACT (Continue on reverse if necessary and identify by block number) <u>See Reverse Side</u>					
20. DISTRIBUTION/AVAILABILITY OF ABSTRACT UNCLASSIFIED/UNLIMITED <input checked="" type="checkbox"/> SAME AS RPT. <input type="checkbox"/> DTIC USERS <input type="checkbox"/>			21. ABSTRACT SECURITY CLASSIFICATION <u>Unclassified</u>		
22a. NAME OF RESPONSIBLE INDIVIDUAL <u>DR. ALAN H. ROSENSTEIN</u>			22b. TELEPHONE NUMBER (Include Area Code) <u>(202) 767-4933</u>		22c. OFFICE SYMBOL <u>AFOSR/NE</u>

Summary

This document represents a final report of the research work conducted at Stanford University under AFOSR Grant 81-0022 from 1 October 1980 to 30 September 1985. Below we give a very brief summary of the most important results of that work. These results are described in more detail in the body of this report and in a complete way in the dissertations and published papers that have come from this research.

The research work conducted under this grant deals with the structure and mechanical properties of high temperature metals and alloys in which a significant contribution to strengthening is made by dispersed second phase particles. Because the research has been conducted on both aluminum and nickel based alloys, this report is organized along these lines.

We have studied, for the first time, the combined effects of oxide dispersion and solute strengthening at high temperatures in an ODS aluminum-magnesium alloy. We find that the oxide particles contribute significantly to the strength at high temperatures, as expected, but that the strength of the ODS alloy at intermediate temperatures is actually lower than that for the solid solution without oxide particles. This suggests that solute strengthening cannot be combined efficiently with oxide strengthening in the design of ODS alloys. Because rapidly solidified aluminum alloys are so similar to ODS aluminum alloys, a study of the high temperature strength properties of Al-Fe-Ce alloys has also been conducted. We find these alloys to be much stronger than ODS aluminum at low and intermediate temperatures but they are much weaker than the ODS alloys at very high temperatures. This weakening effect is caused mainly by twinning deformation of the intermetallic particles at high temperatures. This suggests that much more refractory particles, such as oxides or carbides, are needed for high temperature strength. We have used mechanical alloying to create oxide and carbide particles in Al-Fe-Ce powders and we have found that a very significant improvement in high temperature strength can be achieved in this way. High temperature compressive strengths can be increased by as much as a factor of five using this technique.

We have studied the high temperature deformation and fracture properties of ODS nickel based alloy in both the fine grained and coarse grained conditions. The fine grained condition typically exists just after hot working, but before the final annealing treatment

is used to cause grain coarsening. We find extraordinarily fine grains present in MA 6000 under these conditions, with some grains being as small as $0.25\mu\text{m}$. As expected, the alloy is superplastic in this state and the superplasticity occurs only at very high strain rates. A maximum tensile elongation of 300% can be achieved at 1000 C at a strain rate of almost 1 per second. This suggests that these alloys can be readily formed and shaped in this condition. In the coarse grained condition ODS alloys are extremely creep resistant because the grains themselves are strengthened by oxide dispersoids and because the few grain boundaries that are present are nearly parallel to the stress axis. For this reason ODS alloys typically exhibit a threshold stress, below which the creep rate falls to a very low value. However, at very high temperatures appreciable creep occurs below the threshold stress. We have studied this kind of creep in MA 754 and found that it is associated with grain boundary sliding and cavitation. Unrecrystallized regions of fine grains present along some of the longitudinal grain boundaries permit sliding on these boundaries and cavitation to occur on the transverse boundaries. Resistance to this kind of creep failure process can be improved by designing the alloy with a smaller volume fraction of second phase particles so that the grain coarsening process can go to completion.



Accession for	
NTIS	✓
ERIC	
GA	
JACS	
PER CALL JC	
By	
Distribution	
Availability	
Dist	
A-1	

I. Introduction

It has been well known for a long time that metals and alloys lose their strength at elevated temperatures because diffusional processes lead to coarsening of the microstructure responsible for the strength. Cold worked metals lose their strength at high temperatures because dislocations are able to climb and annihilate. Precipitation strengthened alloys also become weak at high temperatures, both because the precipitates dissolve and because they coarsen by diffusional processes. In order to produce strong metals and alloys at high temperatures it is essential that the strengthening mechanisms be based on strengthening phases that do not degrade at high temperatures. One of the important ways to achieve this is through the use of second phase particles of very stable phases, such as oxides and carbides. Thus oxide dispersion strengthened (ODS) alloys represent a very important class of high temperature alloys. These materials have played an important role in high performance gas turbine engines which require high temperature strength and they are expected to play a significant role as structural materials for hypersonic aircraft. The so-called "Orient Express" airplane will have to be made of very light weight materials that can withstand very high temperatures. It seems certain the metals and alloys will not meet these requirements unless they are strengthened by ceramic phases, such as oxides or carbides in either dispersoid or fiber form. All of the work conducted under this research program relates to the structure and high temperature mechanical properties of these high performance ODS alloys. The work we have done should provide guidelines for the design and use of these materials in advanced aircraft engines and structures.

All of the work presented here is described in much more detail in the dissertations and publications listed at the end of this report. Only the highlights of our findings will be described here. Because the work we have done relates either to high temperature aluminum alloys or to high temperature nickel based alloys, we have organized this report along these lines.

II. Research Report

A. High Temperature Aluminum Alloys

1. The Combined Effects of Oxide Dispersion and Solute Strengthening

Virtually all previous investigations of dispersion strengthening have involved alloys in which the matrix is, or behaves like, a pure metal. Although it is of interest to understand how dispersoids affect deformation of pure metals, in any commercial application dispersion strengthening will be combined with other strengthening mechanisms. At very high temperatures, where dispersion strengthening is most effective, the only other stable strengthening mechanism is that associated with solute, as precipitates tend to dissolve or coarsen at high temperatures and dislocation substructures tend to anneal out. To understand the combined effects of dispersion strengthening and solute strengthening we have studied the high temperature compression strength properties of an Al-Mg solid solution alloy containing oxide dispersoids [1].

That magnesium solute atoms are effective in strengthening aluminum at high temperatures is evident from the data shown in Fig. 1. Here the high temperature creep data for aluminum and aluminum-magnesium solid solution alloys of various compositions are compared. For convenience of comparison the strain rates and stresses have been normalized, respectively, by the lattice self diffusivity and elastic modulus of pure aluminum. It is obvious that the Al-Mg alloys are stronger than pure aluminum by at least a factor of three at all stresses, strain rates and temperatures (the different temperatures are not shown as they are taken into account by the normalizing factors). It is apparent that solute strengthening represents an effective strengthening mechanism at high temperatures.

In order to study the combined effects of solute and dispersion strengthening we have measured the high temperature compression strength properties of IN-9051, an ODS Al-Mg alloy. The results of these tests are shown in Fig. 2, again in normalized form. The data for Al-Mg solid solutions without the dispersoid particles are shown as a solid line. At the highest temperatures the ODS Al-Mg alloy is very much stronger than the

Aluminum and Al-Mg Alloys

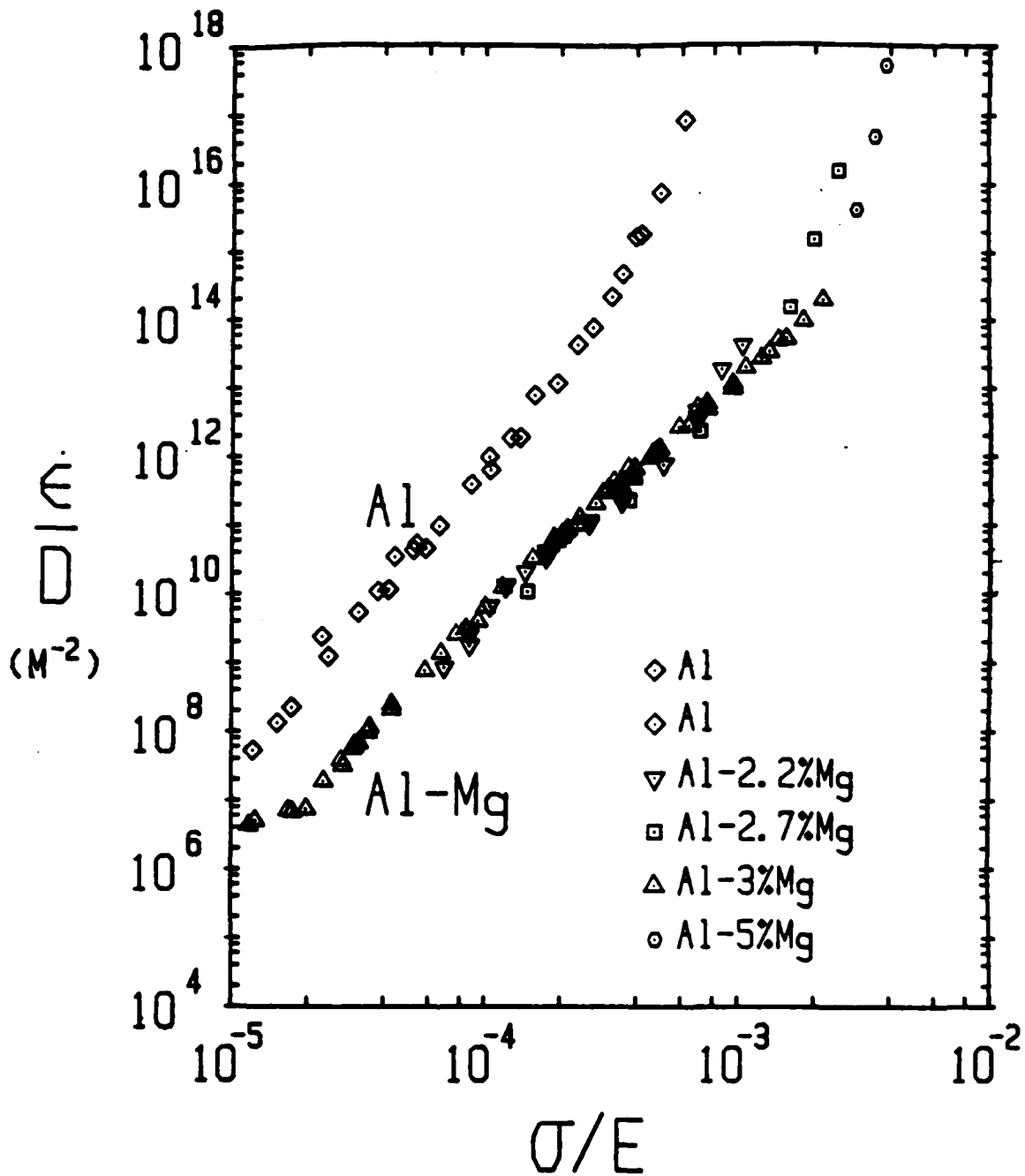


Fig. 1. Temperature compensated steady state strain rate as a function of the modulus compensated steady state stress for Al and several Al-Mg alloys. The diffusion coefficient D is that for self diffusion of Al. The data from the literature were taken from several different sources.

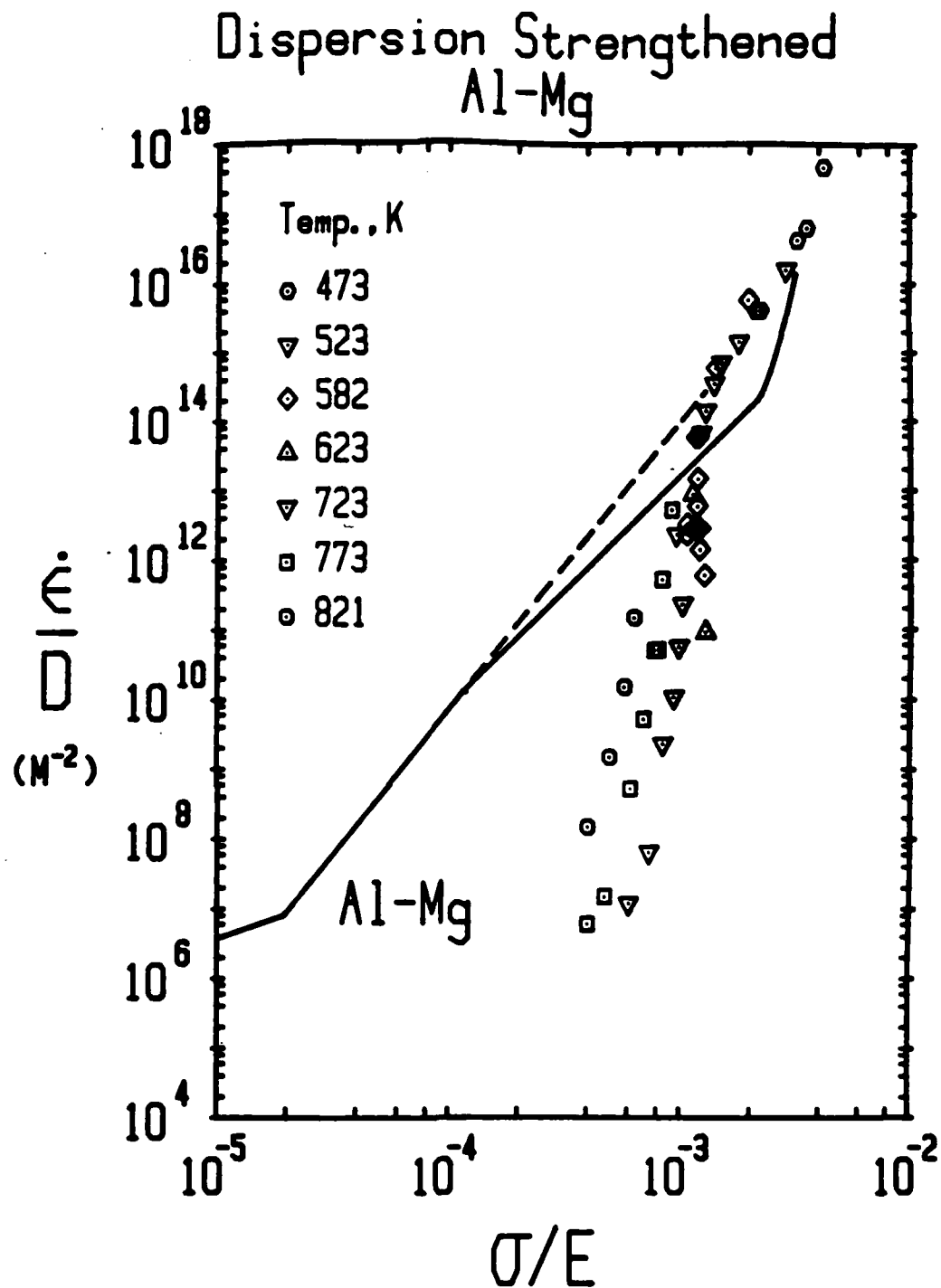


Fig. 2. Temperature compensated steady state strain rate as a function of modulus compensated steady state stress for dispersion strengthened Al-Mg. The solid line shows the steady state creep law for Al-Mg solid solutions without dispersoids. The diffusion coefficient D is that for self diffusion of pure Al.

solid solution, as expected. The dispersoids do increase the high temperature strength of the solid solution. An unusual effect is found at lower temperatures and higher strain rates. Here the ODS Al-Mg alloy is actually weaker than the solid solution without particles.

We have explained the weakening effect of particles by noting that the motion of a dislocation past a particle by the Orowan looping mechanism will create very sharp cusps in the dislocation line and that the dislocation will escape from its solute atmosphere when this occurs. Support for this concept is found in the data for the lowest temperature and highest strain rate. Here, where the solute atmospheres have been stripped away from the dislocations, the strain rate-stress relation is the same as that for the Al-Mg alloys at very high temperatures (see dashed curve). This is expected because solute atmospheres are also not present on dislocations at very high temperatures.

Another important finding of our work is that the high temperature strength of the ODS Al-Mg solid solution is actually less than that of the ODS pure aluminum. This was not expected because it was supposed that the solute strengthening effect of Mg could be added to the dispersion strengthening effect of Al_2O_3 . However, MgO, not Al_2O_3 , is the stable oxide phase in the ODS Al-Mg alloy. Because MgO is much weaker than Al_2O_3 and can be sheared more readily, it is not as effective as a dispersoid. This accounts for the poorer strength properties of the ODS Al-Mg alloy at very high temperatures.

2. Theoretical Studies of Solute Pinning of Dislocations

Our work on the high temperature strength properties of ODS solid solutions required some knowledge about the interaction between solute atoms and dislocations. Although much theoretical work had been done on the binding force between a dislocation and its surrounding solutes, none of the previous studies had considered realistic laws for the distribution of solutes about the dislocation and none had taken account of the finite (non-singular) stresses at the core of the dislocation. Our first work on this subject [2] dealt with the binding force between a singular edge dislocation and a Fermi-Dirac atmosphere. We found for this case that the pinning force exerted by the solute atoms on the dislocation could not be reduced by increasing the temperature. This is contrary to expectation and to general experience. This unexpected result is obtained because a

singular stress field is used in the calculation even for the dislocation core. To correct this problem it was necessary to treat the case of a Peierls-Nabarro dislocation with a Fermi-Dirac solute atmosphere. This was done in our second work on this subject [3]. With this treatment we found that the pinning force is indeed a function of temperature, as expected. The work shows, however, that it is necessary to consider both realistic forms of the solute distribution as well as realistic stress distributions in the dislocation core to give a proper account of the pinning force exerted by solute atoms on a dislocation. This theoretical analysis proved useful in the study of strengthening of ODS Al-Mg alloys described above.

3. High Temperature Strength Properties of Al-Fe-Ce Alloys

During the past few years considerable effort has been devoted to the development of high strength aluminum alloys to replace titanium alloys for a number of high temperature applications. The alloys that have been developed are formed by rapid solidification techniques and depend on a large volume fraction of finely dispersed intermetallic compounds for their elevated temperature strength. Several investigators have shown that one of the most promising alloy systems of this kind is the Al-Fe-Ce ternary system. The high temperature deformation properties of these alloys had not been studied extensively and these properties had not been compared with the properties of ODS aluminum alloys. The aim of our work was to conduct these studies and to make this comparison [4-5].

A consolidated and extruded bar of Al-8.4 wt.%Fe-3.6 wt.%Ce was obtained from Lockheed California and samples were made for compression testing at elevated temperatures. The results of the tests are shown in Fig. 3 in normalized form. Data for ODS pure aluminum is also shown in the figure for comparison. It is evident that the data for Al-Fe-Ce at the lowest temperatures show a high stress exponent for creep flow, much like ordinary dispersion strengthened alloys. Also, at these low temperatures the alloy is much stronger than the ODS Al. However, at high temperatures the Al-Fe-Ce alloy becomes much weaker than the ODS alloy, in spite of the much higher volume fraction of intermetallic phase in the alloy. Much of our research has been devoted to understanding this weakening process.

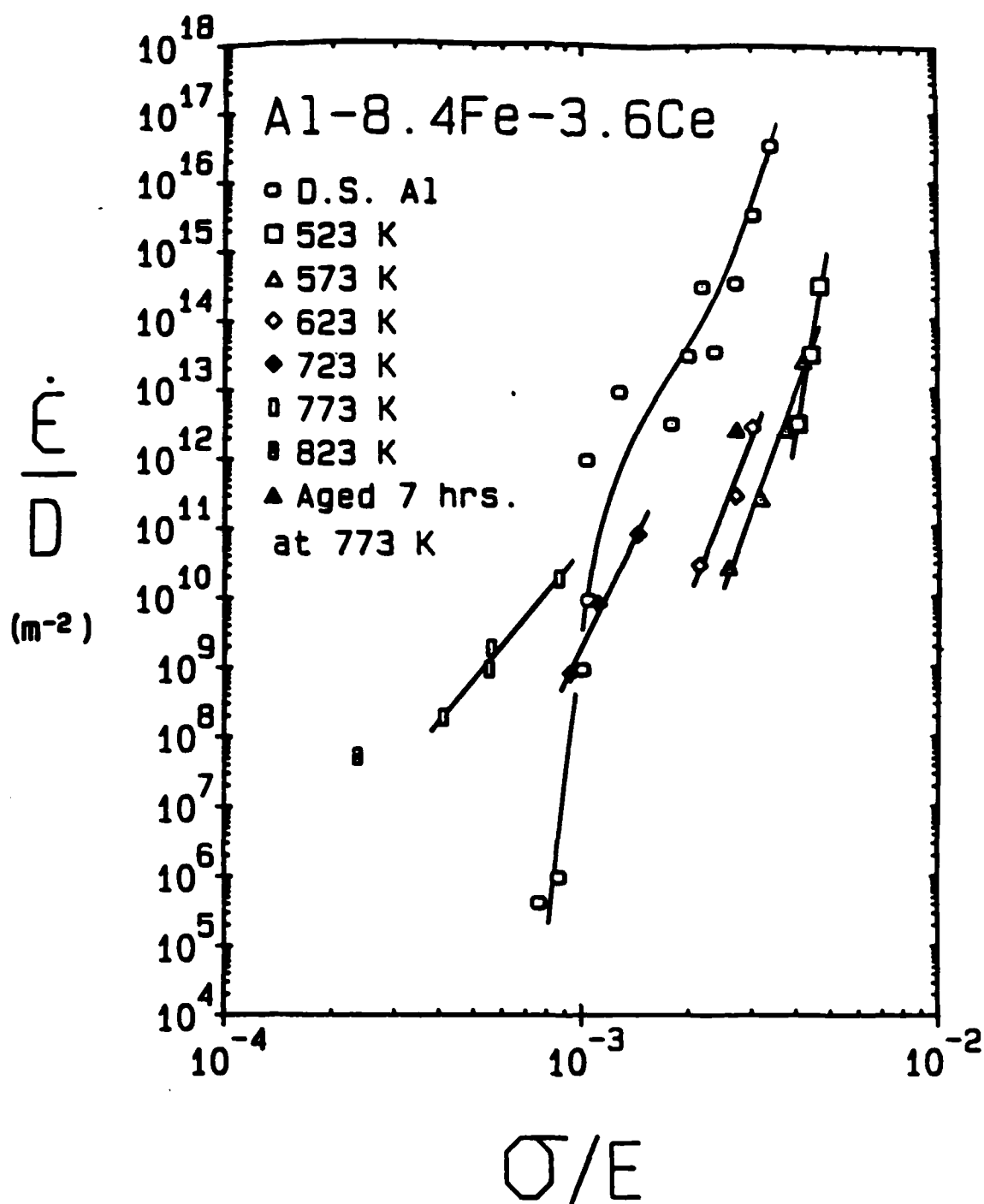


Fig. 3. Temperature compensated strainrate as a function of modulus compensated stress for ODS Al and Al-8.4%Fe-3.6%Ce. The diffusion coefficient D is that for lattice diffusion in pure Al. The modulus E is also for pure Al.

The rapid loss of strength of the Al-Fe-Ce alloy at high temperatures is not caused by coarsening of the intermetallic particles. Some coarsening does occur and we have observed that coarsening but it cannot account for the severe loss of strength that occurs at high temperatures. One sample was aged at 773 K for 7 hours and subsequently tested at a lower temperature (573 K). As shown in Fig. 3, the strength after aging is not much lower than the strength before aging, especially in comparison to the large loss of strength associated with testing at high temperature. This suggests that coarsening is not the primary cause of the weakening.

We believe the severe loss of strength of Al-Fe-Ce at high temperatures occurs because the intermetallic particles begin to deform. Although we have not obtained direct proof of particle deformation, substantial evidence exists to support this claim. We find that the deformation characteristics of Al-Fe-Ce are like those of alloys in which the second phase particles deform and are unlike those of ODS in which the particles do not deform. This is shown as follows. The uncompensated strain rate-stress data for Al-Fe-Ce are shown in Fig. 4. Similar data for a γ' strengthened nickel based alloy (Udimet 115) in which the particles are known to shear during deformation are shown in Fig. 5. For both Al-Fe-Ce and the γ' strengthened alloy the stress exponent decreases with increasing temperature. Also, the activation energy for deformation increases with increasing temperature for both alloys and becomes much larger than that for lattice self diffusion in the matrix. All of these characteristics suggest that creep is controlled by particle deformation at high temperatures, or equivalently that the alloys lose their strength at high temperatures because the particles begin to deform. Figure 6 shows the kind of strain rate-stress relations expected when the particles do not deform. All of the trends in this case are opposite to those found for Al-Fe-Ce. Again this suggests that, unlike ODS alloys, rapidly solidified Al-Fe-Ce alloys lose their strength at high temperatures because the second phase particles begin to deform.

In order to prevent the loss of elevated temperature strength in Al-Fe-Ce it is necessary to supplement the strengthening of the intermetallic phases by the addition of more refractory phases such as oxides and carbides. We have done this by mechanical alloying. Al-Fe-Ce powders have been mechanically alloyed in the presence of an organic lubricant and air. The result is a powder in which oxides and carbides are dispersed in each powder particle on a fine scale. These powders were consolidated by cold pressing and hot extrusion and samples were made for compression testing. Some of the results of

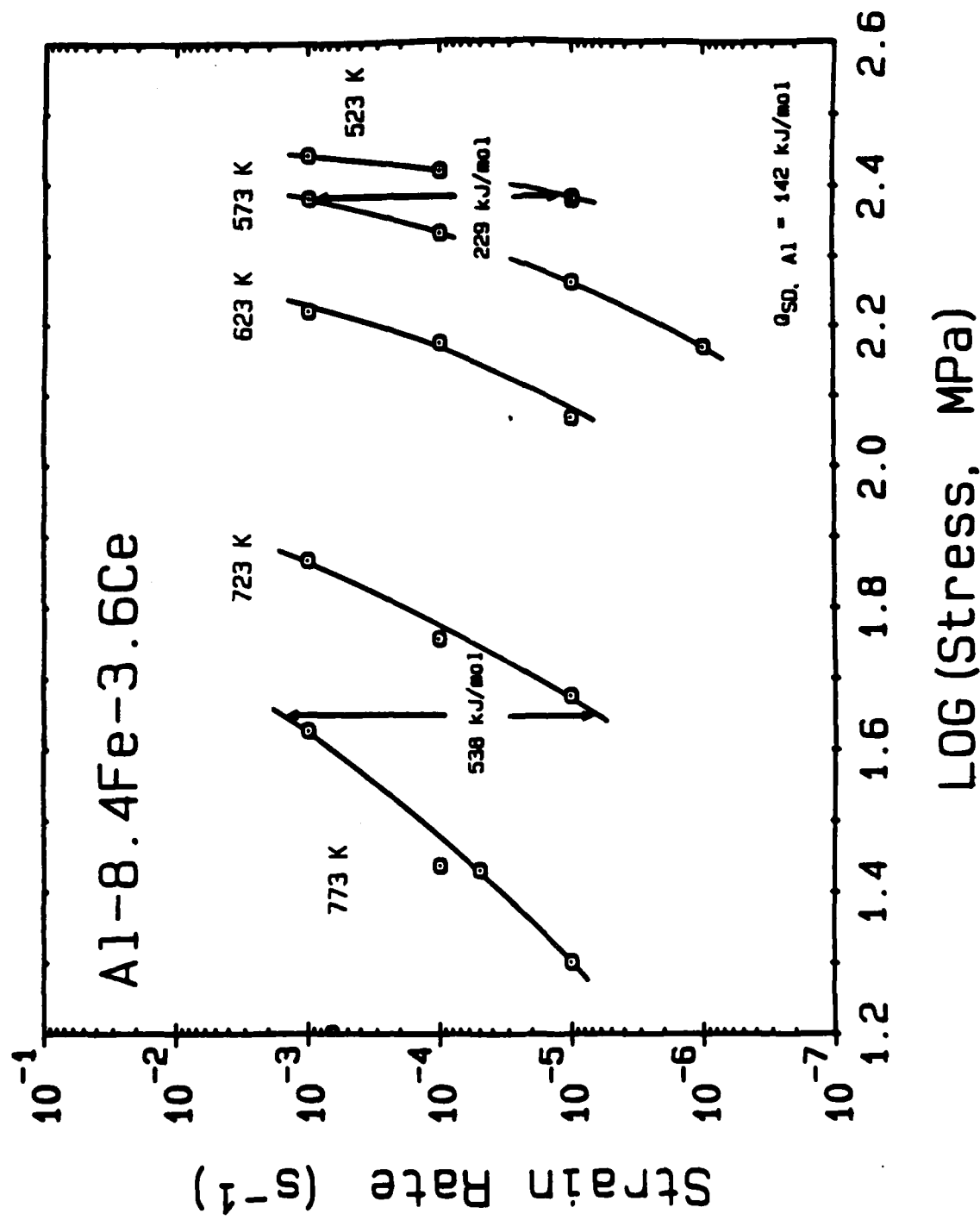


Fig. 4. Double logarithmic plot of strain rate versus stress for as-extruded Al-8.4 wt.%Fe-3.6 wt.%Ce tested in compression. Apparent activation energies for deformation in two temperature ranges shown.

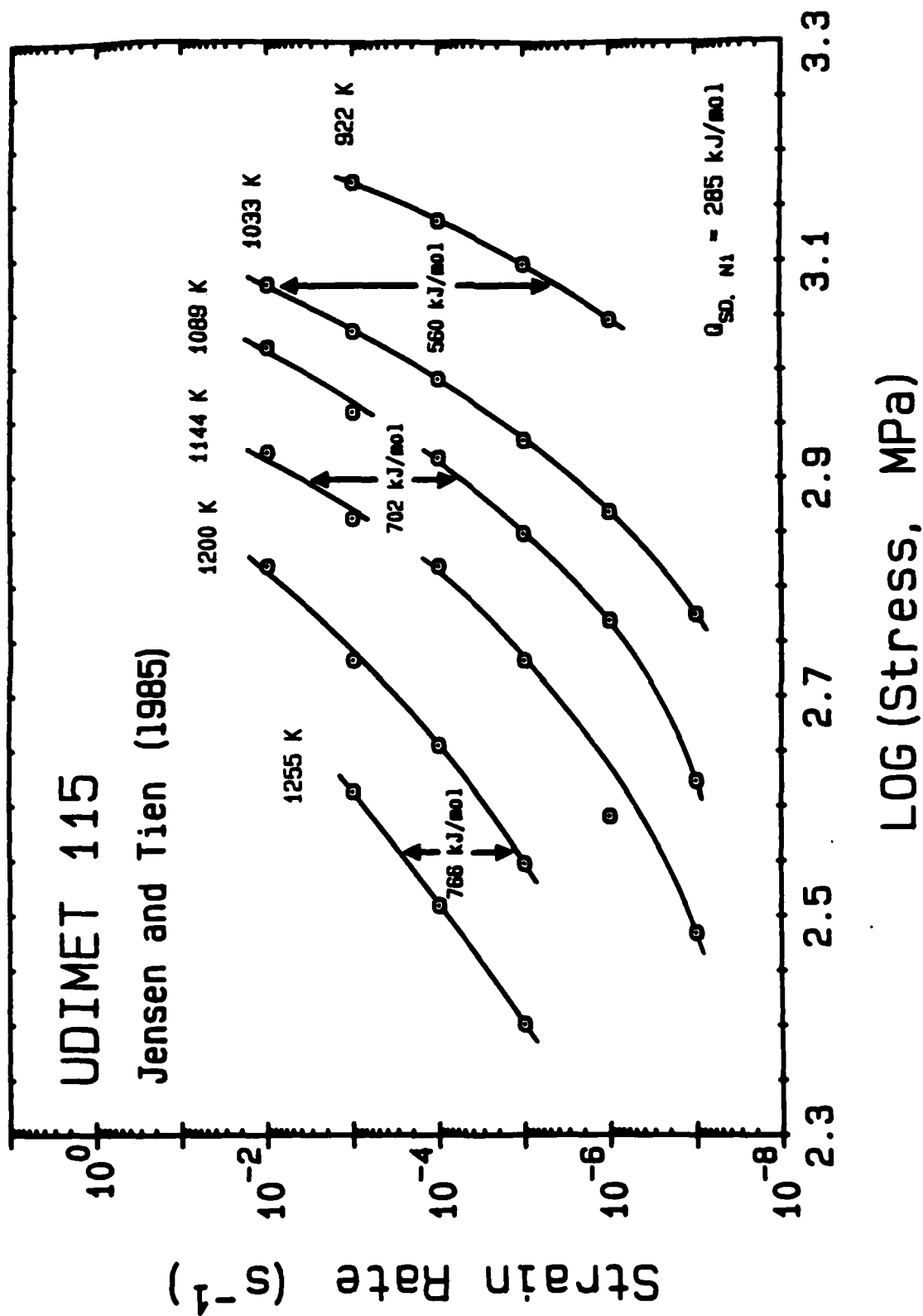


Fig. 5. Double logarithmic plot of strain rate versus stress for a γ strengthened superalloy, Udimet 115. Apparent activation energies for deformation in 3 different temperatures ranges are shown.

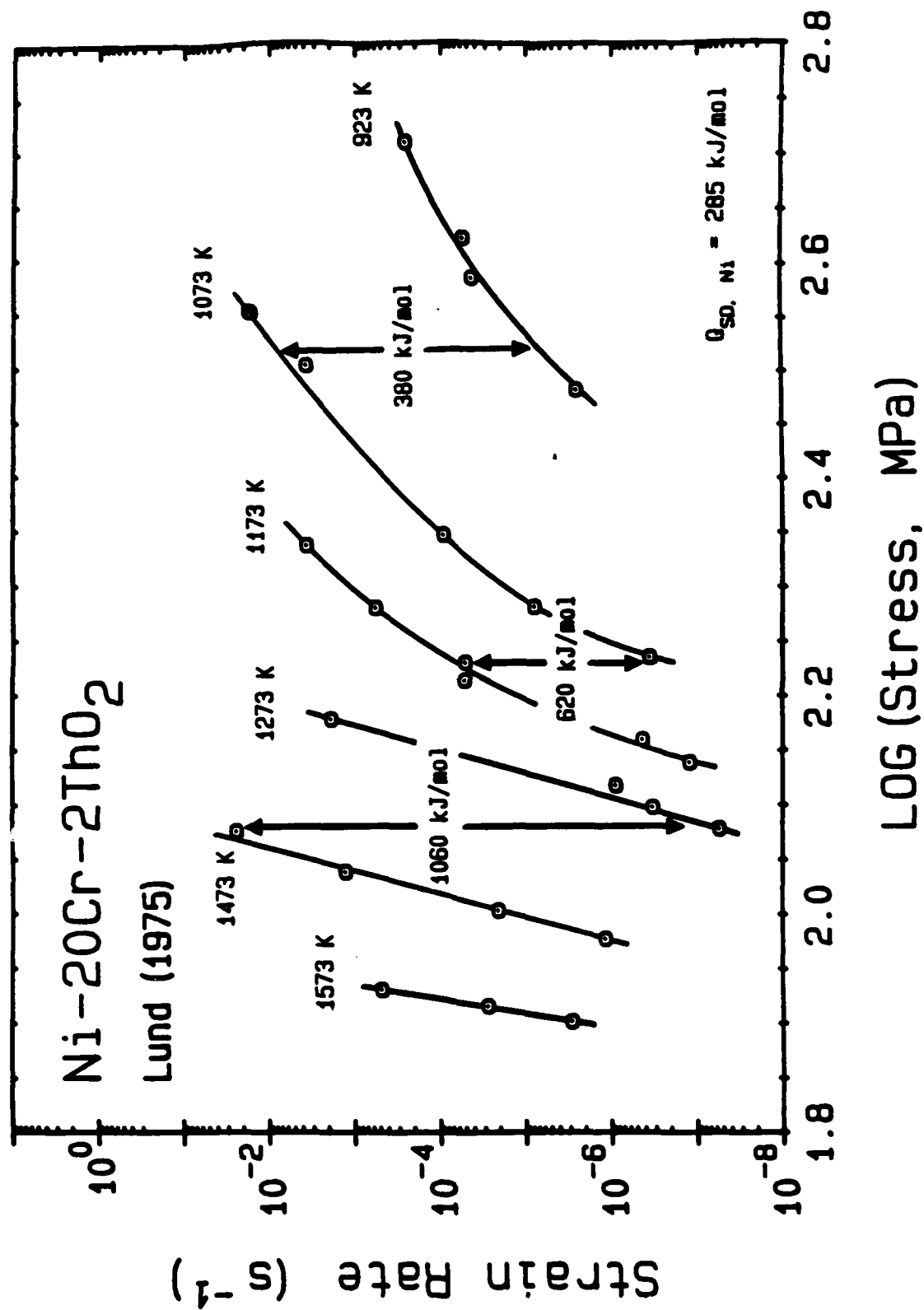


Fig. 6. Double logarithmic plot of steady state creep rate versus stress for single crystal Ni-20 wt. % Cr-2 vol. % ThO₂. Apparent activation energies for creep in 3 different temperature ranges are shown.

the elevated temperature testing are shown in Fig. 7. These data are for materials made of powders that had been mechanically alloyed for 180 minutes. The data for Al-Fe-Ce without mechanical alloying is also shown for comparison. A comparison of the two sets of data indicate that the oxides and carbides present in the mechanically alloyed material improve the strength by as much as a factor of eight ! (773K at 10^{-5} s^{-1}).

B. High Temperature Nickel Based Alloys

1. Superplastic Deformation of ODS Alloys

The synthesis of ODS alloys begins with mechanical alloying of powders to produce the desired composition. Following consolidation of the powders into the form of a billet, the material is hot-extruded and hot-rolled; this produces an extremely fine grain size and a high dislocation density. Finally, the material is zone annealed at approximately nine-tenths of the melting temperature to produce an extremely coarse, elongated grain structure. ODS alloys produced in this way have excellent resistance to creep, but limited creep ductilities. Consequently they cannot be shaped by metal working methods. However, in the hot worked condition, before the coarse grains are formed, the material naturally has much higher ductility. Thus, it may be possible to form intricately shaped parts using ODS alloys in the fine grained condition, and then to obtain coarse grains and high creep resistance by subsequent annealing. In an effort to explore this possibility we have studied both the high temperature deformation characteristics of fine grained MA 754 and MA 6000 and their annealing behavior after deformation [6-7].

The basic deformation properties of fine grained MA 754 and MA 6000 are shown in Figs. 8 and 9. For both alloys, generally high stress exponents for deformation are observed. This is typical of high temperature deformation of ODS alloys. However, both alloys exhibit regimes of deformation behavior in which the stress exponent is quite small (or equivalently that the strain rate sensitivity is quite large). For MA 6000 at 1273 K (1000 C) this occurs at a strain rate of about 1 per second. The strain rate sensitivity at this high strain rate is about 0.5, the value found for many superplastic alloys. Indeed, as shown in Fig. 10, MA 6000 in the fine grained condition does exhibit superplastic

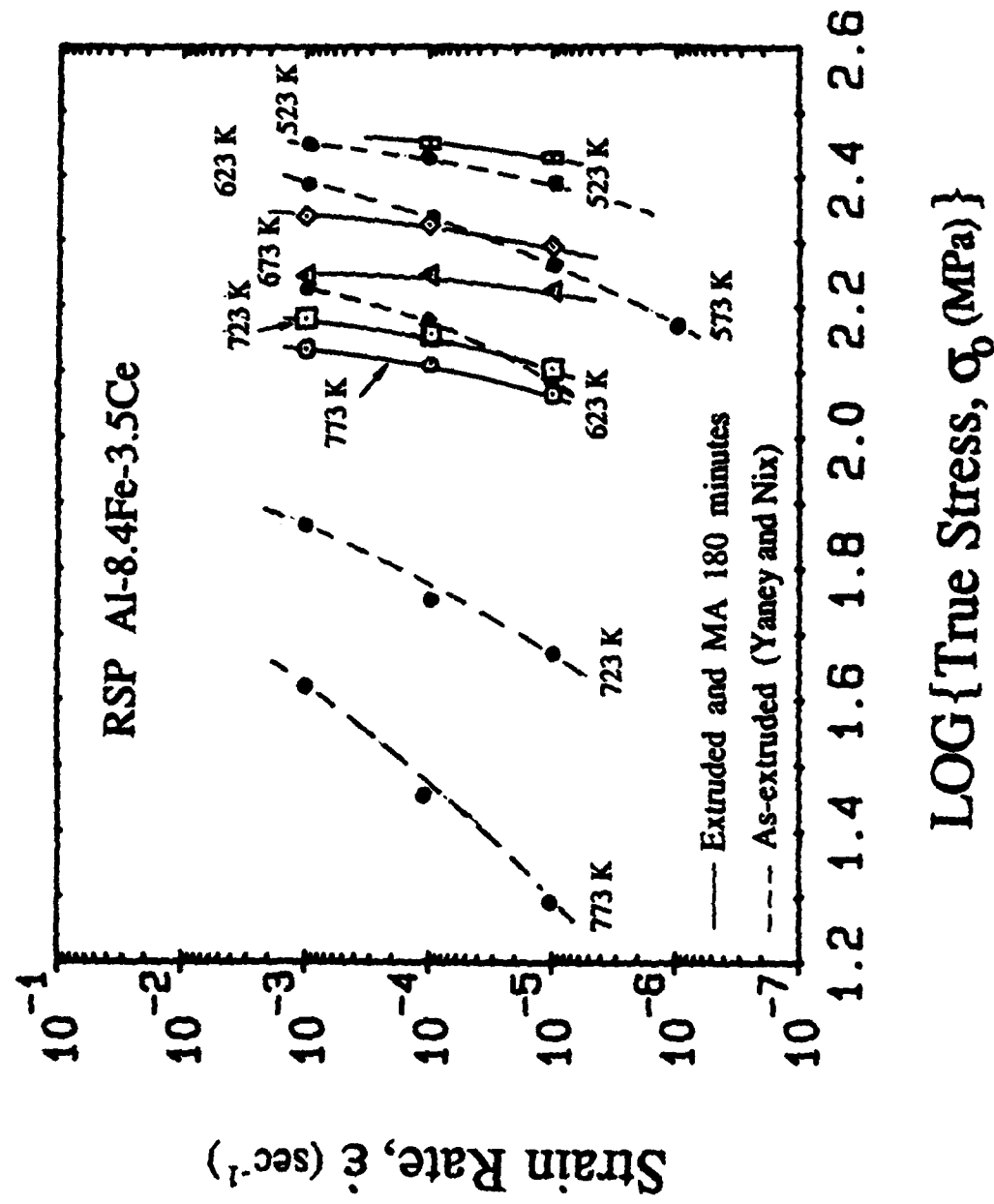


Fig. 7. Strain rate-stress data for Al-Fe-Ce and mechanically alloyed Al-Fe-Ce at various temperatures. The mechanically alloyed material is stronger than the RSP material by as much as a factor 8! at the highest temperature and lowest strain rate.

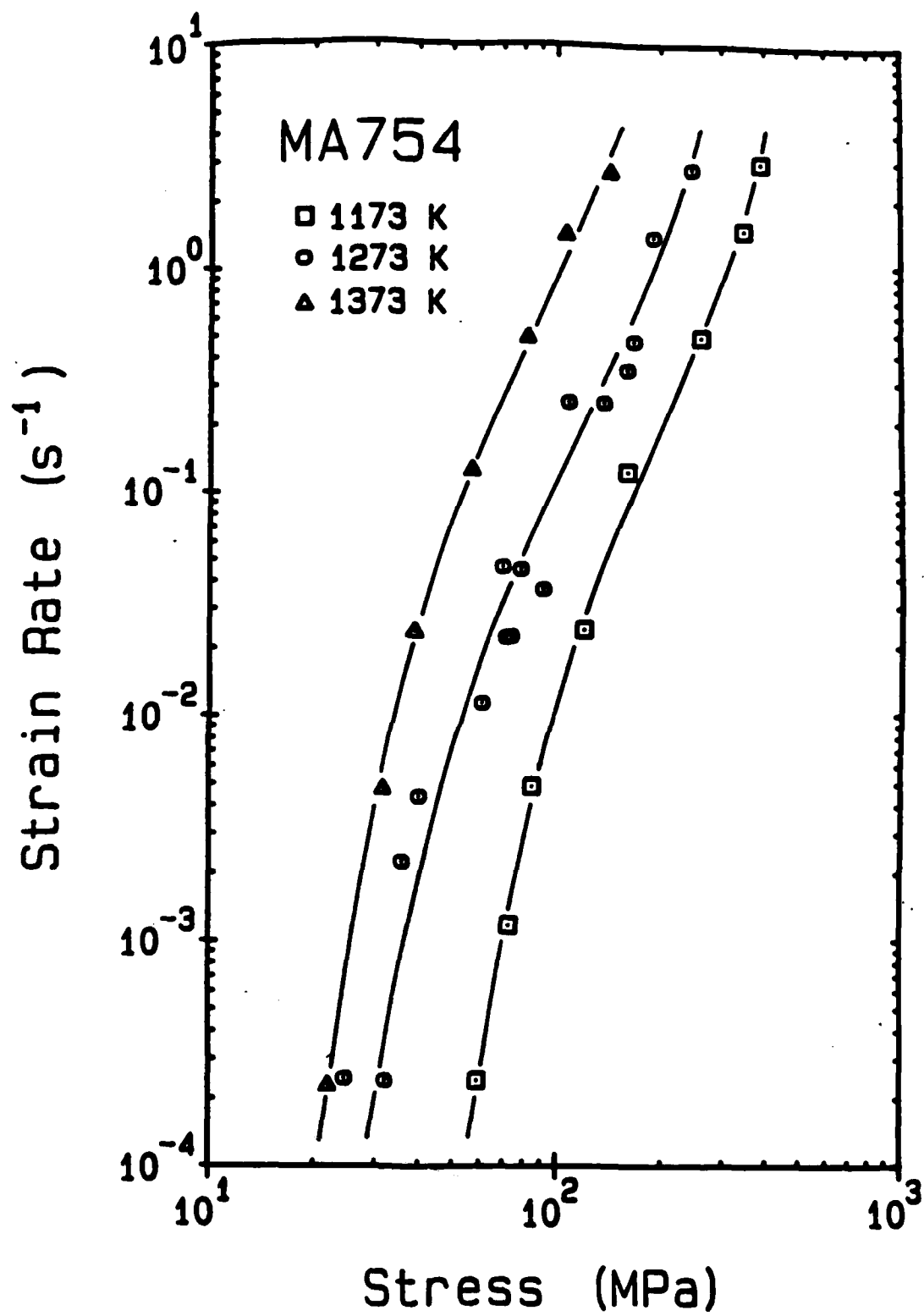


Fig. 8. Plastic strain rate vs flow stress for MA 754.

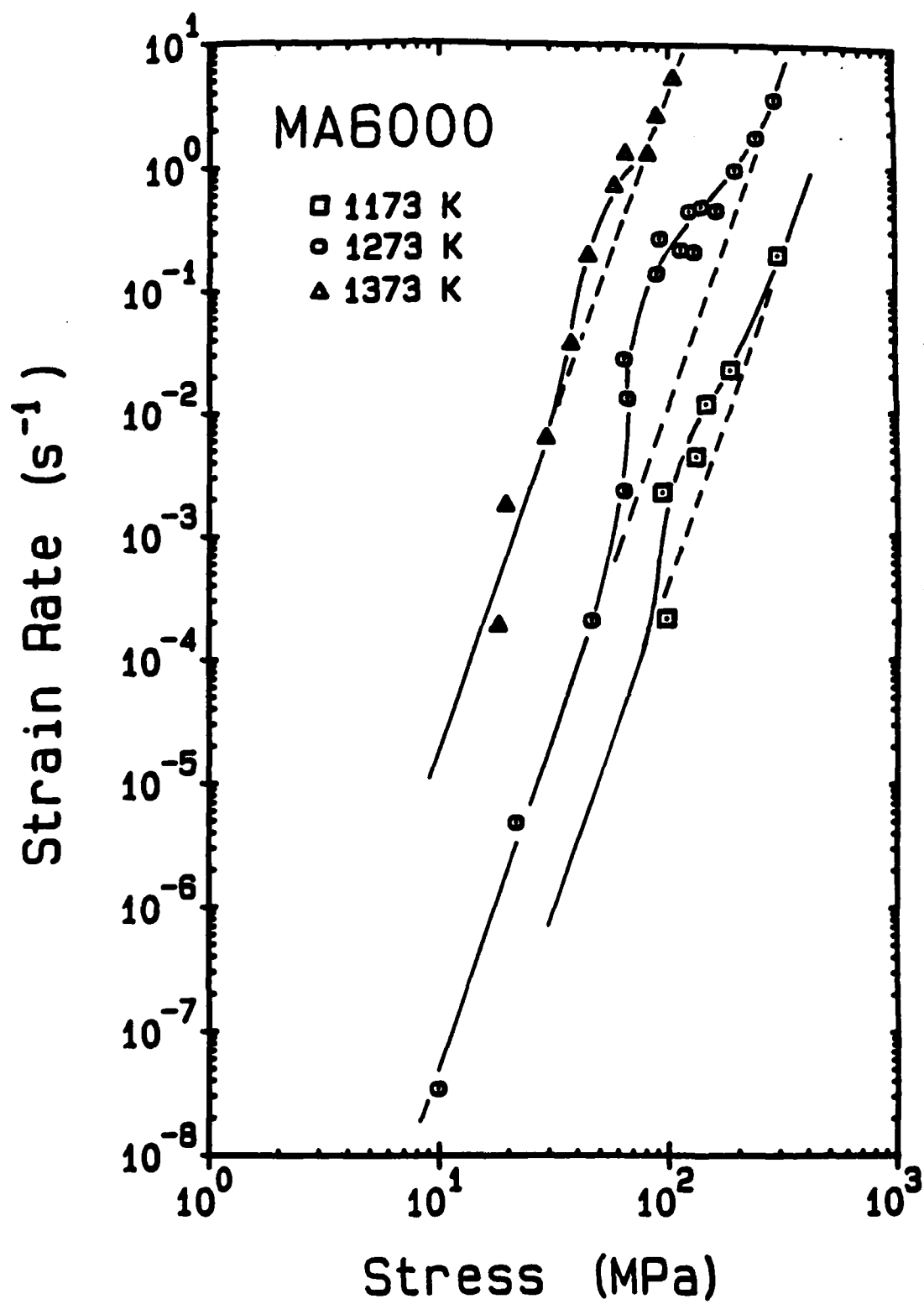


Fig. 9. Plastic strain rate vs flow stress for MA 6000.

MA6000 SPECIMENS DEFORMED AT 1273°K **(FROM GREGORY GIBELING AND NIX REF: 25)**

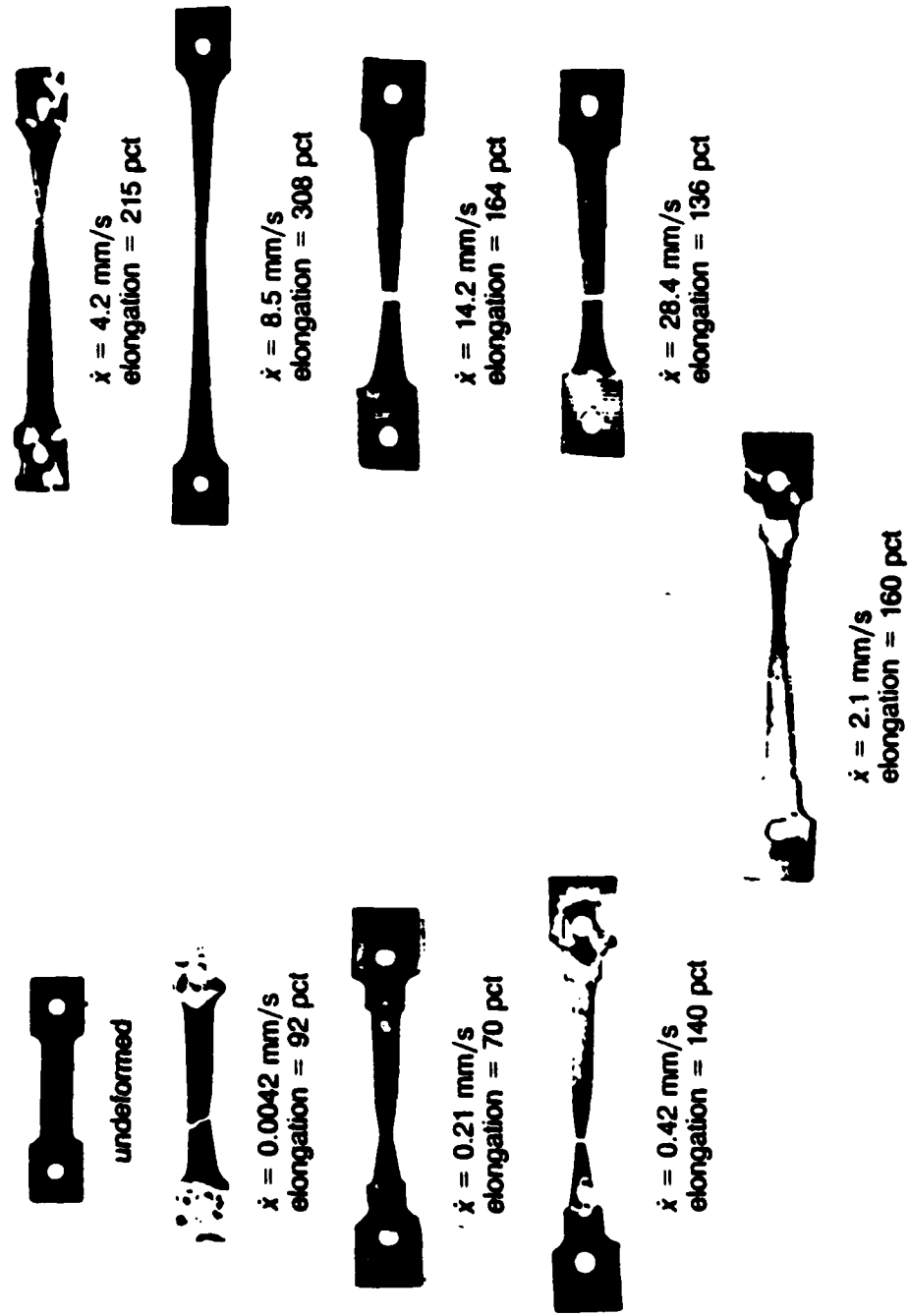


Fig. 10. Appearance of MA 6000 specimens deformed at 1273 K.

behavior under these conditions. These properties indicate that MA 6000 can be superplastically formed at 1000 C. It is of interest to point out that the superplasticity found in MA 6000 occurs at an unusually high strain rate because of the very fine grain size of this material. Superplastic alloys with grain sizes of 2-3 μm can typically be superplastically deformed at a strain rate of 10^{-3} per second. Because the grain size of MA 6000 is an order of magnitude smaller than this, the corresponding strain rate is three orders of magnitude higher (because strain rate varies inversely with the third power of the grain size). Although MA 754 does not show classical superplastic behavior, it does have quite high tensile ductility in the fine grained condition and thus could be deformed extensively in a forming process.

Some understanding of the flow behavior of fine grained MA 6000 can be obtained by considering the results shown in Fig. 11. Here we compare the flow properties at 1273 K with various model predictions. The grain boundary diffusional flow rate (Coble Creep rate) for pure Ni with the grain size of MA 6000 is shown as a dashed line. It is not unreasonable to assume that the Coble creep rate for MA 6000 is slower than that for pure Ni by about an order of magnitude. This is shown as a solid line. We note that this estimate of the diffusional creep rate coincides closely with the data points showing the highest strain rate sensitivity. We believe that the "bump" on the strain rate-stress curve for MA 6000 is caused by a grain boundary diffusional creep process that becomes significant below about 200 MPa. If only grain boundary diffusion were involved, one would expect the creep data to follow the Coble creep law (solid line). Instead what is observed is a threshold stress (shown as a vertical dashed line) below which the creep rate quickly drops to a very low value. We believe that dispersoid particles in the grain boundaries inhibit grain boundary diffusional processes below the threshold stress. At still lower stress ordinary power law creep of the kind expected for pure Ni is observed. It is still not clear why the dispersoid particles should inhibit diffusional processes and not power law dislocation creep.

2. Theoretical Studies of Dislocation Pinning by Oxide Particles

One of the central problems in the theory of dispersion strengthening involves the origin of the threshold stress for creep. It is now well established the dispersion strengthened single crystals can creep at measurable rates only above a critical stress,

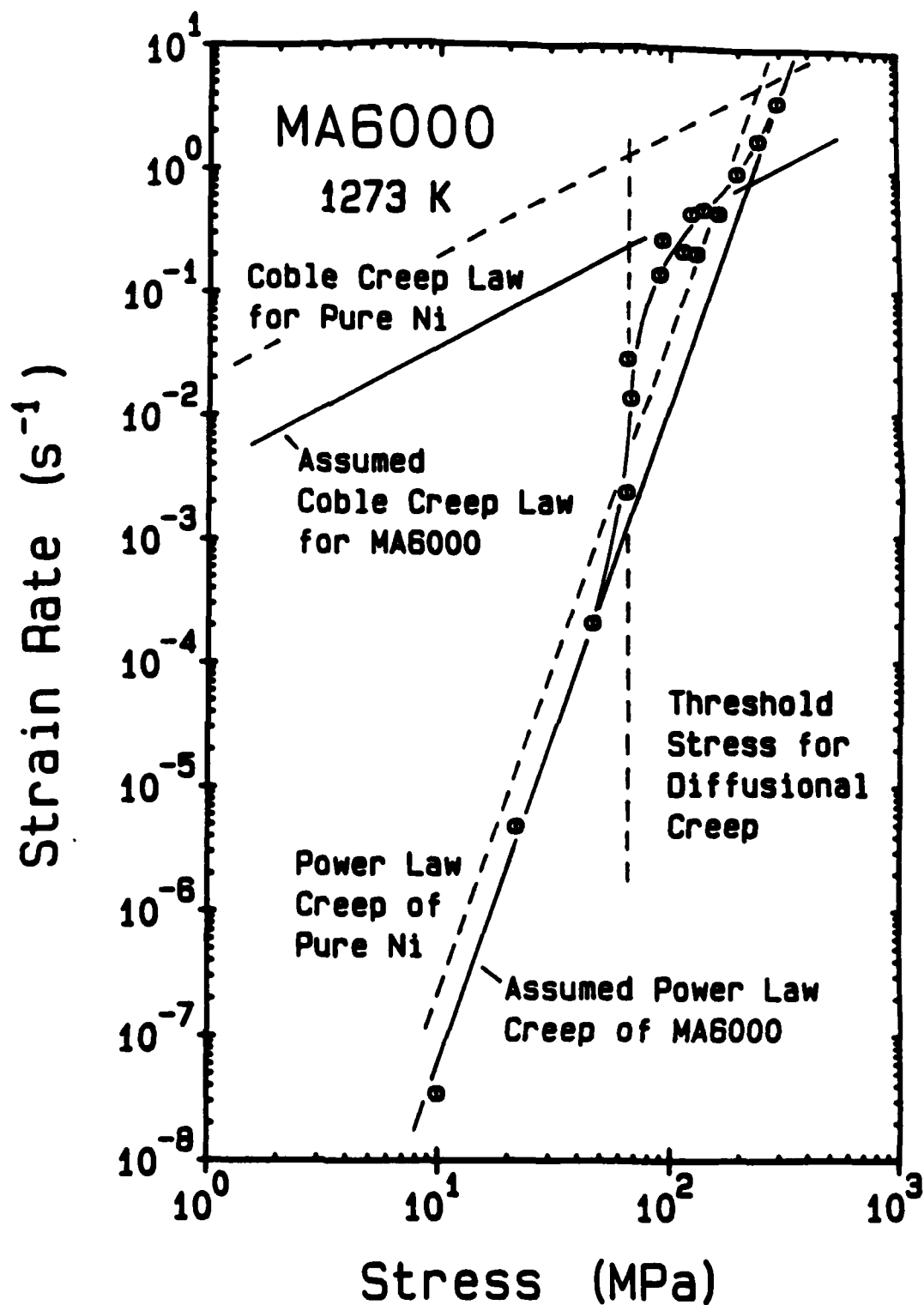


Fig. 11. Comparison of the flow data for MA 6000 at 1273 K with the predicted dislocation and Coble creep behavior of Ni. The assumed dislocation and Coble creep characteristics of MA 6000, as well as the threshold stress for diffusional flow are indicated.

called the threshold stress. However it has been difficult to understand the origin of this threshold stress. Dislocations in the matrix are thought to be repelled by the oxide particles (because the elastic shear moduli of the particles are usually greater than those of typical metal matrices). If this were true, dislocations could easily climb past dispersoids and a threshold stresses would not be observed. We have found that a good account of the threshold stress can be developed if it is recognized the local sliding and diffusional relaxation processes at the interface between the dispersoid and the matrix can cause dislocations to be attracted to the dispersoids [8]. With this picture, dislocations are actually pinned by dispersoids and the threshold stress is that which is needed to pull the dislocations from the pinning particles; the threshold stress is closely related to the Orowan stress. Direct observations of this pinning process have been observed by others.

3. Mechanisms of Creep and Fracture in Grain Coarsened ODS Alloys

There are two guiding principles that govern the design and use of ODS alloys. One involves the strengthening of the grains themselves through the pinning of dislocations by dispersoids and the other involves the minimization of grain boundary sliding and cavitation through the development of a coarse, elongated grain structure. Most previous creep studies have been conducted at stresses and temperatures at which the pinning of dislocations by dispersoids is the controlling factor. Under these conditions creep is characterized by a threshold stress, below which the creep rate falls to a very low value. This implies that at stresses well below the threshold stress the creep rate would be zero, regardless of the temperature. However, the presence of even a few grain boundaries in the sample suggests otherwise. At low stresses and high temperatures grain boundary sliding and cavitation represents an alternative creep mechanism. We have explored this kind of creep behavior by testing two different heats of grain coarsened MA 754 at very high temperatures [9-11].

The materials we have studied have very coarse, elongated grains ($600\mu\text{m} \times 140\mu\text{m} \times 60\mu\text{m}$) with a strong $\{110\}\langle 001 \rangle$ texture. With this kind of grain structure, most of the grain boundaries in the sample are low angle boundaries. Because of this one might not expect much creep to occur by grain boundary sliding. Figure 12 shows that this is not the case. Above 100 MPa the creep stress exponent is very large (46) suggesting that creep is

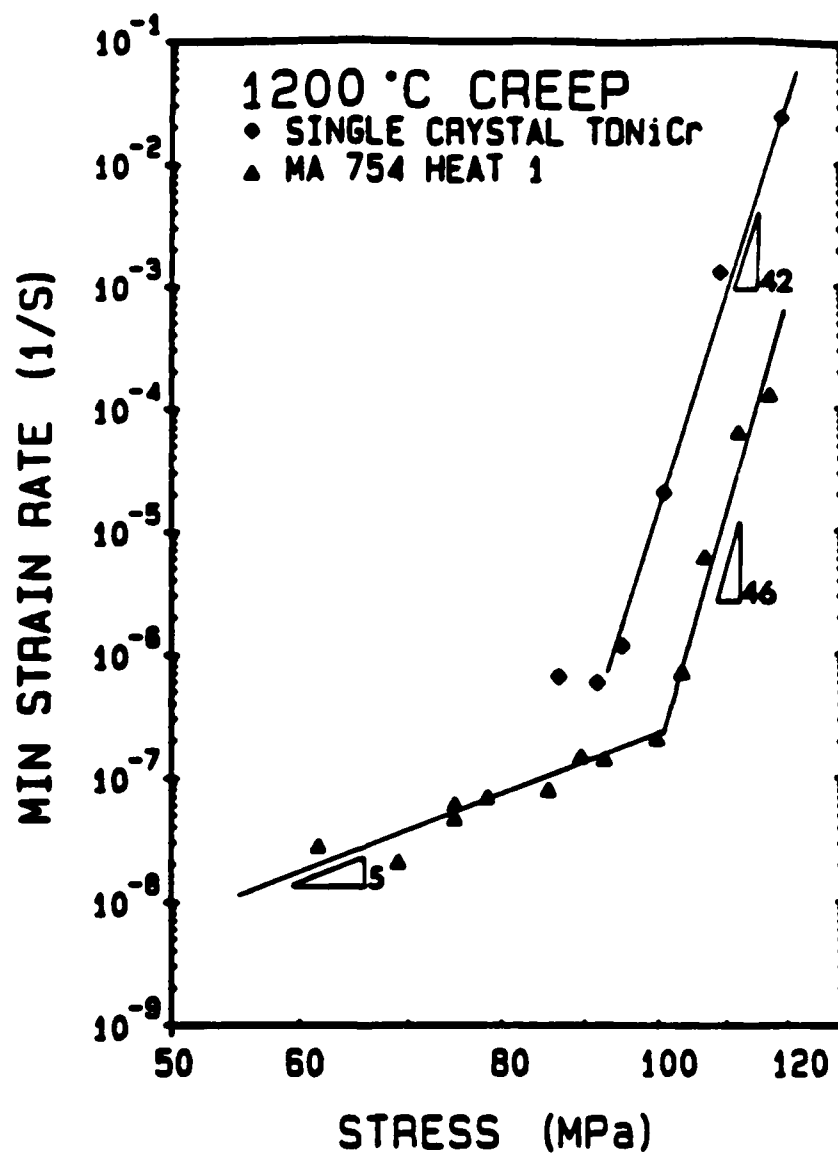


Fig. 12. Minimum strain rate as a function of stress for Heat 1 at 1200 C. Single crystal data ($\langle 001 \rangle$) for TD Ni-Cr are plotted for comparison. At high stresses the stress exponent for Heat 1 is approximately equal to that for single crystal TD Ni-Cr.

controlled by dispersoid pinning of dislocations in this regime. Below 100 MPa, however, a very different picture emerges. Here the creep stress exponent falls to a very low value for a dispersion strengthened material. At low stresses the measured creep rate is many orders of magnitude higher than would be expected from an extrapolation of the high stress behavior. We believe this enhanced creep rate effect is caused by grain boundary sliding and cavitation. We have found that creep fracture is transgranular above the transition stress and intergranular below that stress. This strongly indicates that creep and fracture are controlled by the grains themselves at high stresses and by the grain boundaries at low stresses.

The creep properties of two different heats of MA 754 are shown in Fig. 13. Both heats show a high stress exponent for creep at high stresses indicating that dislocation pinning controls creep at high stresses. Heat 2 is considerably stronger than Heat 1 in this regime. Small angle x-ray scattering studies of these two heats indicate that the dispersoids in Heat 2 are more closely spaced than those in Heat 1 [12]. This accounts for the observed differences in strength. At low stresses and strain rates both heats show lower stress exponents, but the weakening effect in Heat 2 is much more dramatic than that in Heat 1. Indeed, Heat 2 actually becomes weaker than Heat 1 at low strain rates. This remarkable effect is caused by grain boundary sliding and cavitation. The longitudinal grain boundaries in Heat 2 were found to contain pockets of small unrecrystallized grains. We believe that the deformation of these small grains, which is quite rapid at high temperatures, allows the coarse grains to slide past each other and to contribute to the creep rate by pulling apart. Although Heat 1 is weaker than Heat 2 at high strain rates because the dispersoids in it are more widely spaced, it is stronger at low strain rates because the longitudinal boundaries are essentially free of unrecrystallized grains. The comparison of creep properties of Heats 1 and 2 illustrates the trade-off that is always necessary in the design of ODS alloys. A high concentration of dispersoids can produce high strengths at high strain rates (and low temperatures) but it will lead to small, unrecrystallized grains in the structure and this will cause weakening at low strain rates (and high temperatures).

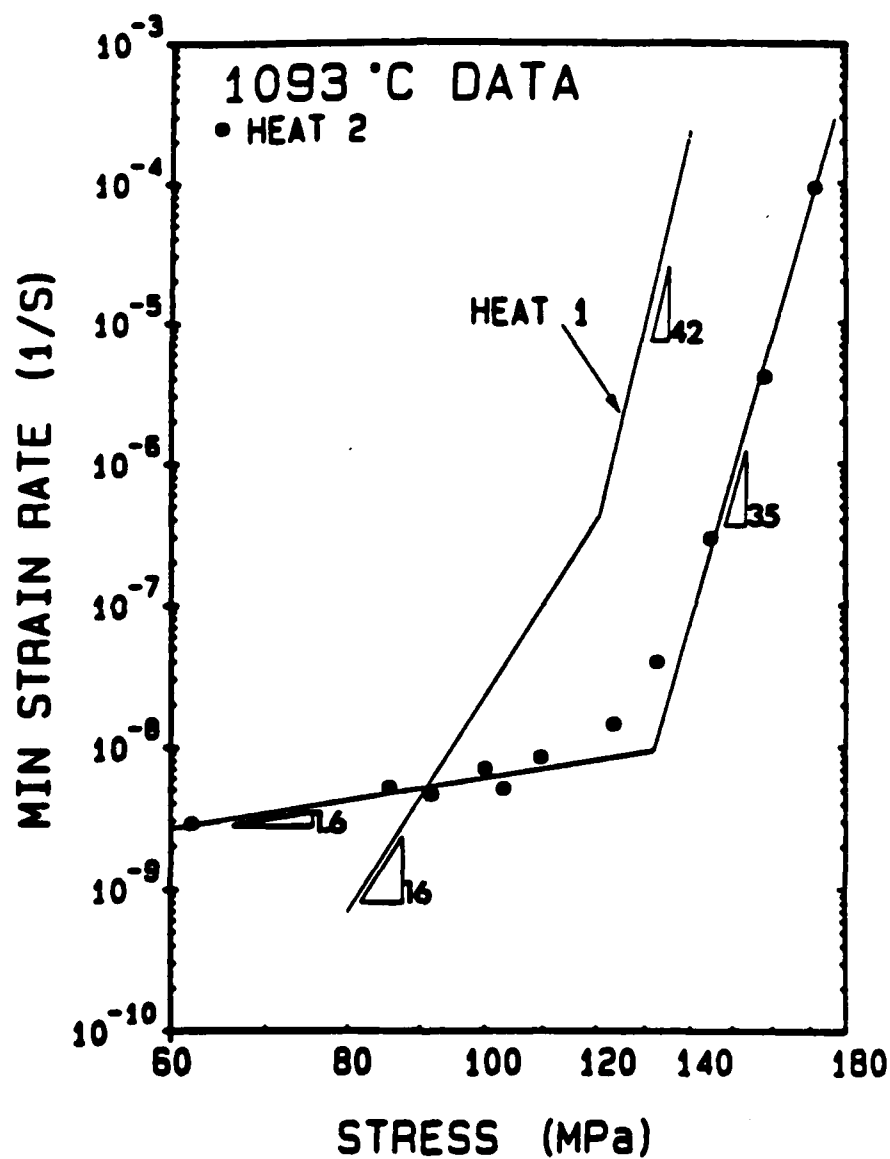


Fig. 13. Minimum strain rate as a function of stress for Heat 2 at 1093 C. The data trend lines of Heat 1 are shown for comparison. The dislocation creep strength of Heat 2 is greater than that for Heat 1. However, the low stress exponent at low stresses for Heat 2 leads to a crossover in creep strength at a minimum strain rate of $5 \times 10^{-9} \text{ s}^{-1}$.

References

1. W.C. Oliver and W.D. Nix, "High Temperature Deformation of Oxide Dispersion Strengthened Al and Al-Mg Solid Solutions", Acta Metall. **30**, 1335 (1982).
2. D.M. Barnett, W.C. Oliver and W.D. Nix, "The Binding Force Between an Edge Dislocation and a Fermi-Dirac Solute Atmosphere", Acta Metall. **30**, 673 (1982).
3. D.M. Barnett, G. Wong and W.D. Nix, "The Binding Force Between a Peierls-Nabarro Edge Dislocation and a Fermi-Dirac Solute Atmosphere", Acta Metall. **30**, 2035 (1982).
4. D.L. Yaney, J.C. Gibeling and W.D. Nix, "Elevated Temperature Deformation and Structural Observations in Al-8.4 Fe-3.6 Ce", Proc. 7th International Conference on Strength of Metals and Alloys, Montreal, Canada, Eds: H.J. McQueen, et al., Pergamon Press (1985) p.887.
5. D.L. Yaney and W.D. Nix, "Elevated Temperature Deformation Behavior of an Al-8.4 wt.%Fe-3.6 wt.%Ce Alloy", (submitted to Metallurgical Transactions)
6. J.K. Gregory, J.C. Gibeling and W.D. Nix, "High Temperature Deformation of Extremely Fine Grained MA754 and MA6000", in Proc. 4th RISQ Int. Symposium of Metall. and Materials Science, 1984, p. 263.
7. J.K. Gregory, J.C. Gibeling and W.D. Nix, "High Temperature Deformation of Ultra-Fine-Grained Oxide Dispersion Strengthened Alloys", Metall. Trans. **16A**, 777 (1985).
8. D. J. Srolovitz, M.J. Luton, R. Petkovic-Luton, D.M. Barnett and W. D. Nix, "Diffusionally Modified Dislocation-Particle Elastic Interactions", Acta Metall. **32**, 1079 (1984).

9. J.J. Stephens and W.D. Nix, "Creep and Fracture of Inconel MA754 at Elevated Temperatures:, Conference Proceedings, Superalloys 1984, October 7-11, 1984, Seven Springs Mountain Resort, Champion, PA., The Metallurgical Society of AIME (1984).
10. J.J. Stephens and W.D. Nix, "The Effect of Grain Morphology on Longitudinal Creep Properties of Inconel MA 754 at Elevated Temperatures", Metall. Trans., 16A, 1307 (1985).
11. J.J. Stephens and W.D. Nix, "Constrained Cavity Growth Models of Longitudinal Creep Deformation of Oxide Dispersion Strengthened Alloys", Metall. Trans., 17A, 281 (1986).
12. J.J. Stephens and S. Spooner, " Determination of Dispersoid Size Distributions in Inconel MA 754 by Small Angle X-ray Scattering", Acta Metall., 34, 303 (1986).

III. Publications Resulting from AFOSR Grant 81-0022

1. D.M. Barnett, W.C. Oliver and W.D. Nix, "The Binding Force Between an Edge Dislocation and a Fermi-Dirac Solute Atmosphere", Acta Metall. **30**, 673 (1982).
2. W.D. Nix, "The Effects of Grain Shape on Nabarro-Herring and Coble Creep Processes", Metals Forum, **4**, 38 (1981).
3. W.C. Oliver and W.D. Nix, "High Temperature Deformation of Oxide Dispersion Strengthened Al and Al-Mg Solid Solutions", Acta Metall. **30**, 1335 (1982).
4. D.M. Barnett, G. Wong and W.D. Nix, "The Binding Force Between a Peierls-Nabarro Edge Dislocation and a Fermi-Dirac Solute Atmosphere", Acta Metall. **30**, 2035 (1982).
5. D. J. Srolovitz, M.J. Luton, R. Petkovic-Luton, D.M. Barnett and W. D. Nix, "Diffusionally Modified Dislocation-Particle Elastic Interactions", Acta Metall. **32**, 1079 (1984).
6. J.K. Gregory, J.C. Gibeling and W.D. Nix, "High Temperature Deformation of Extremely Fine Grained MA754 and MA6000", in Proc. 4th RISO Int. Symposium of Metall. and Materials Science, 1984, p. 263.
7. W. D. Nix, "On Some Fundamental Aspects of Superplastic Flow", 1984 ASM Superplastic Forming Symposium, 1984 WESTEC, Los Angeles, 22 March 1984.
8. J.J. Stephens and W.D. Nix, "Creep and Fracture of Inconel MA754 at Elevated Temperatures", Conference Proceedings, Superalloys 1984, October 7-11, 1984, Seven Springs Mountain Resort, Champion, PA., The Metallurgical Society of AIME (1984).

9. J.K. Gregory, J.C. Gibeling and W.D. Nix, "High Temperature Deformation of Ultra-Fine-Grained Oxide Dispersion Strengthened Alloys", Metall. Trans., **16A**, 777 (1985).
10. J.J. Stephens and W.D. Nix, "The Effect of Grain Morphology on Longitudinal Creep Properties of Inconel MA 754 at Elevated Temperatures", Metall. Trans., **16A**, 1307 (1985).
11. J.S. Wang, J.J. Stephens and W.D. Nix, "A Statistical Analysis of Cavity Nucleation at Particles in Grain Boundaries", Acta Metall., **33**, 1009 (1985).
12. D.L. Yaney, J.C. Gibeling and W.D. Nix, "Elevated Temperature Deformation and Structural Observations in Al-8.4 Fe-3.6 Ce", Proc. 7th International Conference on Strength of Metals and Alloys, Montreal, Canada, Eds: H.J. McQueen, et al., Pergamon Press (1985) p.887.
13. T.G. Nieh and W.D. Nix, "Unloading Yield Effects in Aluminum Alloys", Metall. Trans., **17A**, 121 (1986).
14. A.H. Rosenstein, J.K. Tien and W.D. Nix, "Void Formation in INCONEL MA 754 by High Temperature Oxidation", Metall. Trans., **17A**, 151 (1986).
15. J.J. Stephens and W.D. Nix, "Constrained Cavity Growth Models of Longitudinal Creep Deformation of Oxide Dispersion Strengthened Alloys", Metall. Trans., **17A**, 281 (1986).
16. M.L. Ovecoglu and W.D. Nix, "Characterization Studies of Mechanical Alloying of Rapidly Solidified Al-8.4%Fe-3.4%Ce Powders", Intl. J. Powder Metallurgy and Powder Technology, **22**, 17 (1986).
17. J.J. Stephens and S. Spooner, "Determination of Dispersoid Size Distributions in Inconel MA 754 by Small Angle X-ray Scattering", Acta Metall., **34**, 303 (1986).

18. D.L. Yaney and W.D. Nix, "Elevated Temperature Deformation Behavior of an Al-8.4 wt.%Fe-3.6 wt.%Ce Alloy", (accepted for publication in Metallurgical Transactions)
19. M.L. Ovecoglu, D.M. Barnett and W.D. Nix, "Analysis of the Interfacial Stresses Produced by a Pile-Up of Discrete Edge Dislocations in Two Phase Materials", (submitted to Acta Metallurgica)

IV. Ph.D. Students and Dissertations Supported by AFOSR 81-0022

1. **Warren C. Oliver, Ph.D., Stanford University, 1981.**
"Strengthening Phases and Deformation Mechanisms in Dispersion Strengthened Solid Solutions and Pure Metals"
(currently a Staff Member at the Oak Ridge National Laboratory, Oak Ridge, TN)
2. **Jean K Gregory, Ph.D., Stanford University, 1983.**
"Superplastic Deformation in Oxide Dispersioned Strengthened Nickel Based Superalloys"
(currently a Staff Member at McDonnell-Douglas Inc., St. Louis, MO)
3. **John J. Stephens, Ph.D., Stanford University, 1984.**
"Creep and Fracture of a Yttria Dispersed Nickel-Chromium Alloy"
(currently a Staff Member at Sandia National Laboratories, Albuquerque, NM)
4. **Deborah L. Yaney, Ph.D., Stanford University, 1986.**
"Mechanisms of Elevated Temperature Deformation in Selected Aluminim Alloys and Related Intermetallic Compounds"
(currently a Staff Member at Lockheed Advanced Aeronautics Laboratory, Burbank, CA)
5. **M. Lutfi Ovecoglu (Ph.D. expected, 1986)**

END

11-86

DTIC